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## PROPERTIES OF NIOBIUM RECOVERED FROM MEGABAR DYNAMIC PRESSURES

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### ABSTRACT

Nb was processed and recovered intact from peak dynamic pressures in the range 0.6–1.2 Mbar. Pressure and effective-plastic-strain histories were calculated using a finite-element hydrodynamic computer code. Recovered specimens were characterized by X-ray diffraction, metallography, Vicker's hardness, and superconducting transition temperature  $T_c$ . The maximum change observed in the  $T_c$  of 9.18 K for the unshocked specimen was a decrease of 0.035 K in the specimen shocked to a maximum pressure of 0.6 Mbar. Shock processing of  $V_3Si$  at 1 Mbar was recently reported by Stewart *et al.* to depress  $T_c$  1.8 K from the initial value of 16.4 K. These results indicate that shock-induced defects and disorder have a relatively weak effect on the  $T_c$  of equilibrium phases and suggest that shock-synthesized metastable superconductors might have  $T_c$ 's close to intrinsic values for the ordered material.

### I. INTRODUCTION

High dynamic pressures are known to produce a wide variety of interesting phenomena in condensed matter. For example, metastable stoichiometric Al<sub>15</sub>Nb<sub>3</sub>Si with a high superconducting transition temperature  $T_c$ =18 K has been synthesized only by processing the Ti<sub>3</sub>P phase at Mbar shock pressures.<sup>1</sup> Other specimens of Nb<sub>3</sub>Si with  $T_c$ ≈18 K have also been produced by high dynamic pressure.<sup>2–4</sup> By comparison, the maximum  $T_c$  obtained in metastable Al<sub>15</sub>-phase thin films is about 9 K at a maximum achievable Si concentration of about 20%.<sup>5</sup> The higher  $T_c$  in the shock-synthesized Nb<sub>3</sub>Si is thought to be caused by the higher Si content and smaller lattice parameter. Since the dynamic pressure process has not yet been studied systematically, it is possible that still higher  $T_c$ 's and/or upper critical fields  $H_{c2}$  can be achieved. The  $T_c$  of stoichiometric Al<sub>15</sub>Nb<sub>3</sub>Si has been predicted to be in the range 25–38 K, based on systematics for other Nb compounds.<sup>6–9</sup>

The work we report here was undertaken to develop the experimental and computational techniques to subject materials to shock pressures up to 1.5 Mbar and to recover them for investigations of material structure and physical properties. Our goal is to utilize fast application and release of high densities and temperatures to synthesize new metastable phases. Although most shock processing and synthesis have been done using high explosives,<sup>1-3</sup> two-stage light-gas guns have been utilized,<sup>4,10</sup> as well. We are using a two-stage gun because pressures and loading times are tuneable by selection of impactor material, velocity, and thickness.

We chose to study Nb to investigate the effects of shock-induced defects on the  $T_c$  of an elemental Type II superconductor. The effects of shock-induced defects in Nb are expected to be greatest in specimens processed at Mbar pressures, because Nb has been predicted to have maximum shock-induced hardness at about 0.9 Mbar.<sup>11</sup> Cold rolling Nb at low strain rates ( $\sim 10^{-1}$ /s) causes an increase in  $T_c$  of 0.1 K or less.<sup>12,13</sup> Strong shock waves produce much higher strain rates of at least  $10^8$ /s or larger<sup>14</sup> and might produce different defect structures and have a different effect on  $T_c$ . Of course, the higher strain rates are accompanied by high shock and residual temperatures which can anneal defects.

## II. GENERATION OF HIGH DYNAMIC PRESSURE

Strong shock waves were generated by using a two-stage light-gas gun<sup>15</sup> to accelerate planar, 3 mm-thick steel impactors to velocities in the range 2.5-4 km/s and impacting specimens in a Pb/steel target fixture illustrated in Fig. 1. This fixture was chosen to be sufficiently strong and massive to withstand Mbar shock pressures and to contain the specimen, which flows plastically at late-time. A design which transfers momentum to materials other than the specimen and minimizes plastic flow is clearly very desirable also. In three experiments impact pressures were 0.6, 1.0, and 1.2 Mbar in the Nb specimens and were obtained by measuring impactor velocity  $U_I$  and shock-impedance-matching using the known Hugoniot equations of state for steel and Nb.<sup>16</sup>

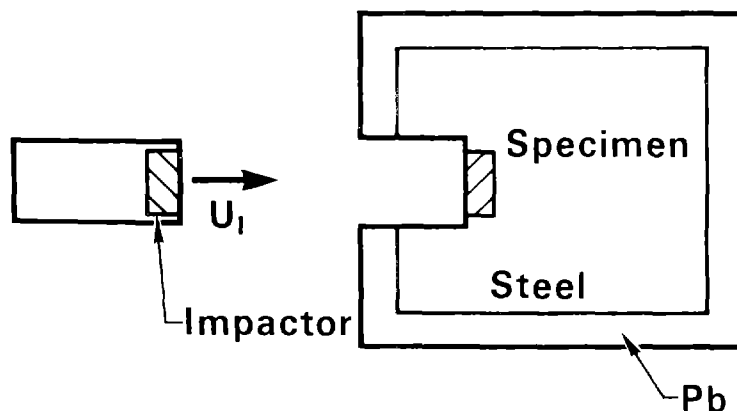


Fig. 1. Illustration of specimen recovery fixture for processing of materials at Mbar dynamic pressures. Strong shock wave is generated on impact of specimen by impactor at velocity  $U_I$ .

In order to characterize the loading histories of the specimens, two-dimensional hydrodynamic calculations were performed using the DYNA2D finite element hydrodynamic computer code.<sup>17</sup> The Nb specimen, Pb/steel recovery fixture, steel impactor and polycarbonate sabot were included. Calculations were performed for each of the three measured impact velocities. Mie-Grüneisen equations of state and pressure- and temperature-dependent yield strengths were used.<sup>18</sup> The calculations matched the observed shape change of the specimen, but only if the yield strength was allowed to decrease with temperature. The pressure dependence of the yield strength has a weak effect on the calculation, presumably because most of the flow occurs at late time when the pressure is reduced substantially from the strong impact-generated shock. The calculation shows an impact-driven initial stress pulse of  $\sim 0.8 \mu\text{s}$  duration, followed by late-time ringing that includes a strong tensile release wave near the axis. Figure 2 is a plot of the effective plastic strain versus time for a zone initially near the half-height and half-radius of the cylindrical Nb specimen. Figure 2 shows that only about 20% of the plastic strain is generated by the impact shock and that it is actually dominated by late-time stress ringing.

### III. EXPERIMENTAL RESULTS

All three Nb specimens were recovered intact from maximum shock pressures of 0.6, 1.0, and 1.2 Mbar, respectively. Initial Nb sample shapes were cylinders 13 mm in diameter and 9 mm thick. Final dimensions were about 20 mm in diameter and 4 mm thick, indicating substantial plastic flow. The Nb was nominally of 99.9% purity. Slow-scan-rate X-ray diffraction was performed on the as-received and 1 Mbar-shocked

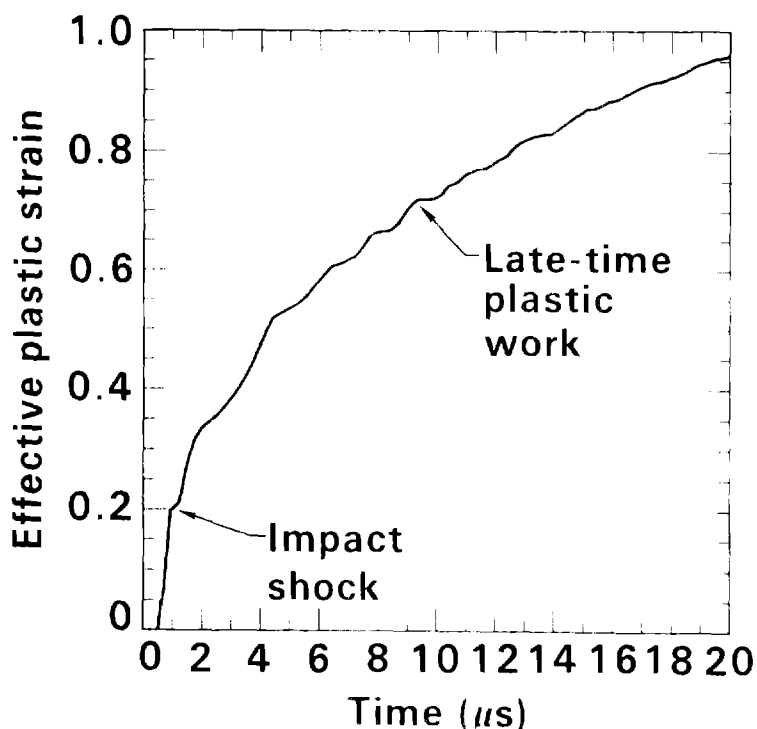


Fig. 2. Calculated effective-plastic-strain history for a zone initially near half-height and half-radius of cylindrical Nb specimen shocked to 1 Mbar.

specimens. Comparison showed a one-to-one matching of the bcc Nb diffraction peaks before and after shocking, with no indication of a shock-induced phase introduction.

Metallographs were obtained for all the specimens and are shown in Fig. 3. The as-received sample has a rather isotropic grain structure in the sense that the long and short dimensions of the grains are comparable. The samples shocked to 1.0 and 1.2 Mbar have pronounced wavy bands of grains, which are long, thin, and oriented preferentially with their long dimensions perpendicular to the shock-wave front. Similar features are observed for Nb cold-rolled to more than 40% deformation.<sup>13</sup>

Vicker's hardness for the as-received and the three shocked specimens are shown in Fig. 4. Pie-sections were cut from each specimen and measurements were made along the radius at the half height of each. The as-received specimen was harder near the outer radius than on axis and permitted investigation of material response as a function of initial hardness. In the center where the Nb was less hard initially, the 0.6 Mbar-shocked specimen is substantially harder than the as-received. Hardness then decreases with increasing  $P_{\max}$ , as for 304 stainless steel and Ni above 0.6 Mbar.<sup>19</sup> Since shock temperature increases with  $P_{\max}$ , these results suggest that increased annealing occurs with increasing shock and residual temperature. At the hardest, outer edge of the specimens, hardness is only weakly dependent on  $P_{\max}$  and the shock processing decreases hardness somewhat. Least-squares parabolic fits through the data indicate broad, weak maxima with radial position, with the largest effect in the 0.6 Mbar-shocked specimen. The prediction of maximum hardness at 0.9 Mbar<sup>11</sup> was based on the assumption of

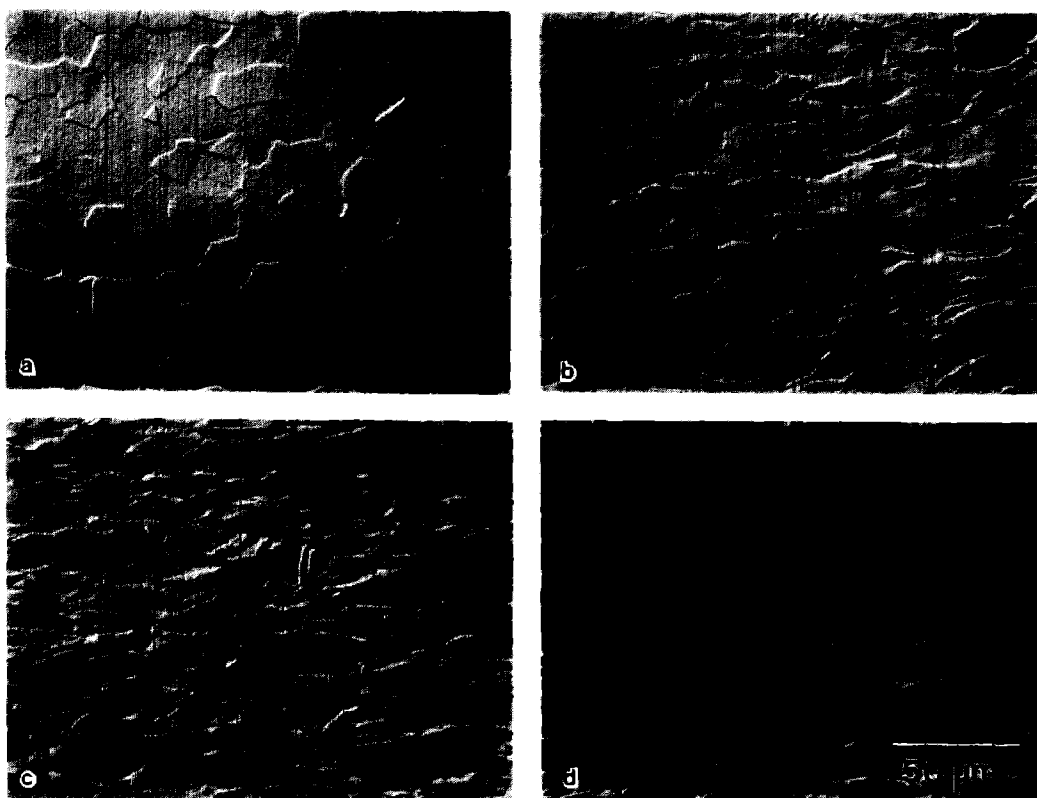


Fig. 3. Metallographs for as-received (a) and specimens shocked to 0.6, 1.0, and 1.2 Mbar (b-d). Metallographs for shocked specimens are oriented such that impact shock traveled in direction from top to bottom.

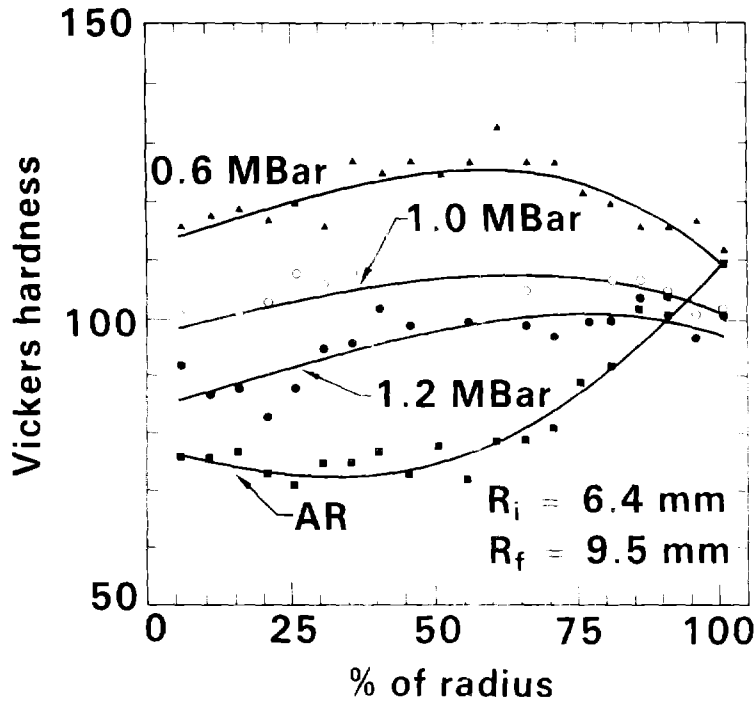


Fig. 4. Vicker's hardness, along half-height of cylindrical specimens, as function of radius for four specimens of Fig. 3. Radii of shocked specimens are about 1.5 times that of unshocked specimens.

negligible plastic deformation, a condition that clearly was not the case here.

The superconducting transition temperatures of the Nb samples were determined by means of ac magnetic susceptibility  $\chi_{ac}$  measurements at 20 Hz in a  $\text{He}^4$  cryostat. The superconducting transition temperature  $T_c$  is defined at the 50% value of the change  $\Delta\chi_{ac}$  in  $\chi_{ac}$  between the normal and superconducting states. The widths of  $T_c$  were taken as the difference between the temperatures for the 10% and 90% value of  $\Delta\chi_{ac}$ . Sections about 1mm thick were cut from the center to the outer radius of the disks and these were then further cut at various radii to obtain the final samples. Up to six samples were measured in a single run; 33 specimens were measured in all from the as-received and three shocked cylinders. For the as-received specimens  $T_c = 9.18 \pm 0.02 \text{ K}$ ; widths of the transitions were 0.01-0.03 K. The maximum decrease in  $T_c$  occurred for the 0.6 Mbar-shocked specimen. This decrease is less than 0.035 K, occurs at 50% of the outer radius and correlates roughly with the weak maximum observed in the hardness data for this specimen at the same radial position.

#### IV. SUMMARY

In conclusion, the  $T_c$  of Nb plastically deformed by maximum shock pressures of 0.6, 1.0, and 1.2 Mbar was found to decrease at most 0.035 K from the value of 9.18 K for the unshocked specimen. Shock processing  $\text{V}_3\text{Si}$  at  $\sim 1 \text{ Mbar}$  depresses the transition temperature 1.8 K from the initial value of 16.4 K.<sup>20</sup> These results indicate that shock-induced defects and disorder have a relatively weak effect on the superconducting transition of equilibrium phases and suggest that shock-synthesized

metastable superconductors might have  $T_c$ 's close to intrinsic values for the ordered material.

The largest decrease in  $T_c$  occurs at a radial position near 50% of the outer radius of the Nb disk shocked to 0.6 Mbar. At this same location the hardness has a maximum. Both these observations are consistent with maximum shock-induced defects at this radial position.

Hydrodynamic computer calculations can now be used to design loading profiles to investigate the response of material structure and properties to different stress histories.

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